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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : A61L 9/00	A2	(11) International Publication Number: WO 98/32473 (43) International Publication Date: 30 July 1998 (30.07.98)
(21) International Application Number: PCT/IB98/00063 (22) International Filing Date: 16 January 1998 (16.01.98) (30) Priority Data: 197 03 711.9 23 January 1997 (23.01.97) DE (71) Applicant (for all designated States except US): AIR LIQUIDE SANTÉ DEVELOPPEMENT [FR/FR]; 10, rue Cognacq Jay, F-75007 Paris (FR). (72) Inventors; and (75) Inventors/Applicants (for US only): BEILFUSS, Wolfgang [DE/DE]; Timmkoppel 39, D-22339 Hamburg (DE). GRADTKE, Ralf [DE/DE]; Lindenweg 19, D-25436 Tor- nesch (DE). MANGOLD, Herbert [DE/DE]; Furchenacker 24, D-22523 Hamburg (DE). (74) Agent: CONAN, Philippe; L'air Liquide S.A., 75, quai d'Orsay, F-75321 Paris Cedex 07 (FR).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>Without international search report and to be republished upon receipt of that report.</i>
(54) Title: REDUCTION OF EMISSIONS OF VOLATILE COMPOUNDS BY ADDITIVES (57) Abstract This application relates to the use of isothiazolone compounds, mercapto compounds, cyclodextrin compounds, iodopropynyl butylcarbamate, diiodomethyl p-tolyl sulphone, zinc oxide, hydrogen peroxide or mixtures of two or more such compounds as additive for the reduction of emissions of volatile compounds which are potentially hazardous or cause unpleasant odour from compositions which comprise constituents which form such compounds which are potentially hazardous or cause unpleasant odour.		

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Reduction of emissions of volatile compounds by additives

The invention relates to the use of special additives for reducing the emission of volatile compounds from compositions and products which comprise constituents which can form or release such volatile compounds. In particular, the invention relates to the provision of additives for the reduction of emissions of volatile compounds which are potentially hazardous or cause unpleasant odour, such as HCN, amines and thio compounds.

There are many substances and compositions which form or release volatile compounds which have unpleasant odour and are potentially hazardous on accumulation. Depending on the extent of the release or emission of volatile compounds, the unpleasant odour or the potential hazard occurs in a range which extends from "not perceptible" or "not present" to "predominant". In particular, a decrease of the emission is desirable in the case of substances or compositions which release the compounds in question to a relatively great extent. However, even in the case of substances and compounds which release these substances in amounts which are very small and are basically negligible, so that in the case of correct handling, there is no question with regard to odour pollution or safety hazard, a decrease in emissions can also be desirable, however, in order to keep the safety margin, until the individual MAK values are reached, as high as possible.

For example, there are compositions from which, under certain conditions, compounds such as HCN, amines such as NH_3 and CH_3NH_2 or thio compounds such as H_2S , for example, are released. In this case, a decrease in the release, in particular, of HCN is of interest, which can proceed in highly variable extents from, for example, cyanide or thiocyanate ions or compounds having cyanide or thiocyanate substituents or compositions comprising such compounds.

In addition to the inorganic compounds, in particular, organothiocyanates, which include aromatic and

aliphatic organothiocyanates, are potential HCN sources. Examples of aliphatic and aromatic organothiocyanates are thiocyanate-substituted thiazoles, 3-thiocyanato-N-octylacrylamide and methylenebisthiocyanate (MBT). They frequently have a characteristic unpleasant odour and, under certain conditions, also release volatile compounds such as, for example, thio compounds and HCN. The emission values in this case are, at least in the case of approved commercial products, within the permitted limit values (MAK values), but, in principle, can also lie above these. Emissions of this type and, in particular, elevated emissions, represent a potential hazard to persons handling or using the products containing these constituents.

The contents of the individual compounds in question in the surrounding atmosphere serve, for example, as measured parameter for the emission of a product, e.g., for the emission of HCN, the HCN content in the surrounding air serves as indicator, in which case this can be determined using an appropriate test tube (e.g. a Dräger tube: prussic acid 2/a (hydrogen cyanide) (CH 25701)).

Quantitative determination of emissions such as HCN is comparatively complex and frequently cannot be carried out in practice. To remedy this, therefore, subjective organoleptic tests, which give highly meaningful results, are currently customarily also carried out. In this case, the odour impression of the tested compositions is determined, a reference sample being tested in conjunction.

The object therefore underlying the invention is to reduce emissions of volatile compounds such as HCN, thio compounds and amines, for example, which are potentially hazardous or cause unpleasant odour, from compositions and products which comprise constituents which form such compounds which are potentially hazardous or cause unpleasant odour, and, in this regard, to provide low-emission compositions.

This object is achieved by means of the fact that isothiazolone compounds such as N-octylisothiazolone and benzisothiazolone, mercapto compounds, cyclodextrin

compounds, iodopropynyl butylcarbamate and diiodomethyl p-tolyl sulphone, zinc oxide, hydrogen peroxide, or mixtures of two or more such compounds, are used as additive for the reduction of emissions of volatile compounds which are potentially hazardous or cause unpleasant odour from compositions which comprise constituents which form such compounds which are potentially hazardous or cause unpleasant odour.

Preferred embodiments are the subject-matter of the subclaims.

According to the invention, it has been found that emissions which are potentially hazardous or have unpleasant odour may be greatly reduced or eliminated by addition of the abovementioned additive compounds. The extent of the emission reduction may be set via the concentration of the additives. In the case of products which already comply with the legally prescribed limit values (MAK values), according to the invention, a further reduction or elimination of such emissions can increase the safety margin until the MAK values are reached, whereas in the case of substances and compositions whose emission lies above this, a decrease or elimination of the emissions can lead to the corresponding commercial products being approved or being able to be put on the market.

The reduction can appear in advantages with respect to odour versus a comparison product which contains no additive according to the invention. The advantage achieved is, for example, a reduction of the odour strength and/or a change of the odour note or type of odour.

The following description, to illustrate the invention, is essentially concerned with reducing the emission of HCN from organothiocyanate compounds. However, the invention is not restricted thereto, as the further qualitative results for emissions of thio compounds show. To illustrate the reduction of HCN emissions, the test substances selected were firstly an aromatic thiocyanatothiazole or 3-thiocyanato-N-octylacrylamide and secondly aliphatic methylenebisthiocyanate (MBT).

Suitable emission-reducing additives according to the invention are, for example, the following isothiazolones and mixtures of the same: N-octylisothiazolone, 5-chloro-N-methylisothiazolone, N-methylisothiazolone, a mixture of
5 5-chloro-N-methylisothiazolone and N-methylisothiazolone, 1,2-benzisothiazolone, 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one. A suitable mercapto compound is, for example, Pyrron disulphide. Suitable cyclodextrins are, for example, α -, β - or γ -cyclodextrin (cyclohexaamylose,
10 methylcycloheptaamylose or cyclooctaamylose). In addition, iodopropynyl butylcarbamate, diiodomethyl p-tolyl sulphone, zinc oxide and hydrogen peroxide are suitable.

The preferred emission-reducing additive is N-octylisothiazolone. In the examples, the embodiments
15 according to the invention prepared are preparations which comprise the emission-reducing additive, in particular an isothiazolone, which reduces the emission of the preparations and, inter alia, also acts as an HCN trap.

The invention makes it possible to reduce the release
20 of HCN both in aqueous systems over a wide pH range, such as pH 1 to pH 12, e.g. by adding water-soluble isothiazolones, such as N-methylisothiazolone, benzisothiazolone or 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one, zinc oxide and hydrogen peroxide, and in lipophilic media, e.g. by
25 adding N-octylisothiazolone.

According to the invention, therefore, in particular HCN emissions can be reduced from primarily liquid products (solutions, dispersions, preferably aqueous dispersions).

Since substances and compositions having high HCN
30 emissions are not suitable for the market, according to the invention, the possibility is provided of reducing their emissions so greatly that marketing is thus possible. Moreover, according to the invention, by adding the emission-reducing additives, emissions which are already acceptable
35 and comply with current safety regulations can further be decreased, for example the organoleptic properties can also be improved, which can lead to an increased market acceptance.

Furthermore, it is important that the action, e.g. a biocidal action, ascribed to the treated compositions is not impaired by the emission-reducing additives.

5 The invention can be used, for example, in the area of solutions or dispersions for a fungicidal and algistatic finishing of materials and technical preservation. Concrete areas of application and user products are, for example, preservatives, disinfectants, impregnating agents, paints, sizings and adhesives, primers, coatings, lubricants and
10 plasters.

The emission-reducing additives according to the invention are used at least in a total amount of 0.01% by weight, preferably 0.01 to 10.0% by weight, in particular 0.1 to 7.5% by weight, and preferably 0.5 to 5.0% by weight,
15 based on the composition. When a plurality of emission-reducing compounds are used, the proportion of each of these additive components can be set according to the specific product requirements.

Experimental results are given below which indicate
20 the HCN emission from aromatic organothiocyanate-containing compositions or which confirm the emission-reducing action of the said additive compounds.

Aromatic organothiocyanate + HCN trap

25

Example I

Aqueous dispersions which comprised 7.8% by weight of aromatic organothiocyanate (in the form of 26% by weight of a
30 30% strength dispersion), based on the weight of the dispersions, were admixed with various amounts of different cyclodextrins (0.5%, 1.0%, 2.0%, 3.0% and 5.0%, based on the weight of the dispersions, of cyclodextrin (CD) alpha (Example Ia) or CD beta (Example Ib) or CD gamma (Example Ic)
35 and were tested for compatibility and stability. The procedure and the results of "HCN" measurements are reported below.

Appearance after storage at room temperature for 12 days or 10 weeks:

The dispersions containing CD alpha and gamma (Examples Ia and Ic) were unchanged with respect to homogeneity and yellowish-white colour. In the case of the dispersions containing CD beta (Example Ib), slightly inhomogeneous, unstable brownish dispersions formed, in particular at the higher concentrations.

10 Result:

Cyclodextrin alpha and cyclodextrin gamma lead to homogeneous aromatic-organothiocyanate-containing and comparatively stable compositions. Cyclodextrin beta impairs the stability and homogeneity of aromatic-organothiocyanate-containing compositions.

Example II

Aqueous dispersions which comprised 7.8% by weight of aromatic organothiocyanate (in the form of 26% by weight of a 30% strength dispersion), based on the weight of the dispersions, were admixed with various amounts of N-octylisothiazolone (45% strength solution) (0.5%, 1.0%, 2.0%, 3.0% or 5.0%, based on the weight of the dispersion) and were tested for compatibility and stability. The procedure and results of "HCN" measurements are reported below.

Appearance after storage at room temperature for 7 days:

After 7 days, homogeneous yellowish-white dispersions were present unchanged.

Example III

HCN measurements using a Dräger test tube for HCN (see above) (5 strokes) for the dispersions of Examples I and II:

In this test, the Dräger test tube was used together with a pump. Both tips of the tube were broken off and one end of the tube was then inserted tightly into the pump. The

HCN content was tested by holding the other end of the tube in the air space over the surface of the respective sample in a 50 ml wide-neck sample vial which was filled with 50 g of the sample. The temperature was 23.5°C. The sample vials had been sealed for 4 weeks. For measurement, the air was sucked through the tube. The measurement period was about 60 seconds for 5 strokes and correspondingly about 24 seconds for 2 strokes. The result was then determined on the basis of the coloration (reaction of HCN with HgCl_2 and methyl red). The measurement method corresponds to the Dräger instruction 234-257 of 1995.)

Blank value (without additive, aromatic organo-thiocyanate-containing aqueous dispersions) → approx. 1.5 ppm.

15

Example Ia: + 5 % CD alpha → approx. 1.5 ppm

Example Ib: + 5 % CD beta → 1 - 1.5 ppm

Example Ib: + 5 % CD gamma → approx. 1.5 ppm

Example II: + 5 % N-octylisothiazolone → 0 ppm

20

(45 % strength solution)

HCN measurements using Dräger tubes for HCN (see above)
(5 strokes) for the dispersions according to Example II in a
250 ml wide-neck bottle which contained approximately 40 g of
the respective dispersion:

25

Blank value: approximately 1 - 2 ppm

All other samples: no HCN detectable!!

30 Result:

An addition of cyclodextrin somewhat decreases the HCN emission of aromatic-organothiocyanate-containing dispersions.

35

An addition of N-octylisothiazolone (45 % strength solution) significantly decreases the HCN emission of aromatic-organothiocyanate-containing dispersions.

Example IV

Aqueous dispersions were produced which comprised 7.8% by weight of aromatic organothiocyanate (in the form of 13% by weight of a 60% strength dispersion) based on the weight of the dispersions. The dispersions were homogeneous and whitish-yellow (very high quality).

Example V

HCN measurements using Dräger tubes (5 strokes) of the dispersions of Example IV (aromatic organothiocyanate without and with 1.0%, 2.0%, 3.0% and 5.0% N-octylisothiazolone (45% strength solution):

The dispersions were measured after storage at room temperature for 24 hours in a 250 ml wide-neck bottle which contained approximately 100 g of the respective sample:

	Zero value	After 1 week at room temperature
Blank: without additive	>> 30 ppm	> 30 ppm
+ 1% Kathon 893	approx. 25 ppm	8 ppm
+ 2% Kathon 893	approx. 10 ppm	5 ppm
+ 3% Kathon 893	approx. 5 ppm	2-3 ppm
+ 5% Kathon 893	approx. 2 ppm	2 ppm

The dispersions remained unchanged with respect to homogeneity and whitish-yellow colour. There was a marked odour improvement with respect to the blank value.

The aromatic organothiocyanate content of the dispersions containing 0 or 1% N-octylisothiazolone (45% strength solution) was:

without N-octylisothiazolone	7.1%
with 1% N-octylisothiazolone	7.1%

Result:

An addition of N-octylisothiazolone significantly decreases the HCN emission of aromatic-organothiocyanate-containing dispersions. The odour of such dispersions is improved in this case. There is no decomposition of the organothiocyanate active compound.

Example VI

Solutions containing aromatic organothiocyanate + N-octylisothiazolone:

Liquid preparations based on aromatic organothiocyanate (60 % strength solution) and N-octylisothiazolone (45 % strength solution) (data in parts by weight), HCN emission measurement (see above):

N-octylisothiazolone (45 % strength solution)	0	90	80	70	60	50	40	30	20	10
Organothiocyanate (60 % strength solution)	100	10	20	30	40	50	60	70	80	90

HCN emission in ppm, measured using Dräger tubes at 22°C, measurement vessel 100 ml wide-neck bottle filled with 100 g of solution:

After 48 h	>> 30					< 1	< 1	< 1	< 1	2
After 96 h		< 1	< 1	< 1	< 1					
After 1 week						< 1	< 1	1	1	2

The solutions had not changed after storage at room temperature for 2 months.

Example VII**Solutions containing aromatic organothiocyanate +
N-octylisothiazolone:**

5 Liquid preparations based on aromatic organo-
thiocyanate (60% strength solution) and N-octylisothiazolone
(45% strength solution) (data in parts by weight), HCN
emission measurement (see above):

N-octylisothiazolone (45 % strength solution)	8	6	4	2
Organothiocyanate (60 % strength solution)	92	94	96	98

10 HCN emission in ppm, measured using Dräger tubes at
22°C, measurement vessel 100 ml wide-neck bottle filled with
100 g of solution:

After 48 h 1-3 4-5 17 > 30*

After 1 week 2-3 5 7 17-18

* 2 strokes

15 **Result:**

By adding N-octylisothiazolone to aromatic-
thiocyanate-containing solutions, homogeneous mixtures which
have a significantly decreased HCN emission are obtained in
all mixing ratios.

20 **Further additives decreasing the HCN emission of aromatic
organothiocyanate**

25 In the studies, further HCN traps were found, where
in each case homogeneous dispersion comprising 7.8% by weight
of aromatic organothiocyanate was prepared containing 2% by
weight of active compound of the respective additive (in the
case of H₂O₂ (30% strength) it was therefore 6.66% by
weight), and the HCN emission was measured as a function of
time using a Dräger tube.

30 HCN emission in ppm, measured using Dräger tubes at
22°C, measurement vessel 50 ml wide-neck bottle filled with
25 g of dispersion:

HCN trap	HCN after 48 h in ppm	after 1 week ppm	pH after 1 week
Without addition	> 30	20	4.5
A) Cyclodextrin alpha	20	20	4.5
B) Cyclodextrin beta	25	20	4.5
C) Cyclodextrin beta	25	20	4.5
D) Cyclodextrin gamma	25	20	4.5
E) Zinc pyrithione	25	25	4.7
F) 1,2-Benzisothiazolone	5	< 1	4.6
G) Pyrion disulphide	8	1-2	4.2
H) N-Methylisothiazolone	2-3	2	4.0
I) Iodopropynyl butylcarbamate	10-12	10	4.6
J) Hydrogen peroxide (30%)	10	7	2.7
K) Zinc oxide	6	8	7.1
L) Benzothiophene-2-cyclohexyl- carbamide S, S-dioxide*	20	20	4.1
M) 5,6-Dihydro-2-methyl-2H-cyclo- pent(d)isothiazol-3(4H)-one**	0	0	4.1
N) PVP	25	25	4.0
O) PVP + hydrogen peroxide (30%)***	18	720	2.5

* 50% active content, initial concentration = 4%

** 50% active content, initial concentration = 4%

5 *** 12% H₂O₂; initial concentration = 16.6%

Result: The further additives studied lead to a reduction of the HCN emission, which reduction, in comparison with the isothiazolones, is somewhat lesser.

10 Preferred examples of these further additives lead to a comparatively high reduction (to < 20 ppm) of the HCN emission of aromatic-organothiocyante-containing compositions.

15 HCN emission and stability of commercial compositions which comprise aromatic organothiocyante and N-octylisothiazolone:

A test was made of whether instabilities or reductions in active compound occur during storage of

commercial compositions which comprise aromatic organo-thiocyanate and N-octylisothiazolone.

Result:

5 An addition of N-octylisothiazolone to, for example, film preservatives based on compositions which contained aromatic organothiocyanate, led to optically clear, homogeneous solutions. The HCN emission was significantly reduced. The active compound content is virtually unchanged
10 after storage at room temperature or +40°C for 1 month.

The same applies to the HCN emission of pure acrylic masonry paint which was treated in a corresponding manner.

Result:

15 At the application concentration, no HCN is detectable and the stability was excellent.

Example VIII

20 3-Thiocyanato-N-octylacrylamide:

Preparation of 3-thiocyanato-N-octylacrylamide and N-octyl-3-thiocyanatoacrylamide from N-octylisothiazolone (NOITZ, extracted from N-octylisothiazolone (45 % strength solution) by shaking with toluene and water) and KCN

25 213 g (1 mol) of NOITZ + 65 g (1 mol) of KCN were stirred together in 600 ml of water. 10% strength H₂SO₄ was added dropwise with stirring and cooling (maximum temperature 30°C). The mixture was stirred for 3 hours. The precipitate was filtered off by suction, taken up in 500 ml of warm DIPE,
30 dried over Na₂SO₄ and crystallized out with addition of approximately 1.5 l of PE. The crystals were filtered off by suction and washed with PE. After drying, 145.4 g of N-octyl-3-thiocyanatoacrylamide (60.5 % yield) resulted, in the form of yellowish crystals, F: 81°C.

35 The 3-thiocyanato-N-octylacrylamide which was isolated in crystalline form is virtually insoluble in water (< 0.01 %) and dissolves at about 2.5 to 10% in glycols or glycol ethers. The 2.5 to 10% strength solutions of

3-thiocyanato-N-octylacrylamide in glycols or glycol ethers emit HCN, that is to say approximately 30 ppm (1 stroke) after storage at room temperature for 1 week, whereas the crystalline material had an HCN emission of approximately 25 ppm at 5 strokes (Dräger tube, see above).

Solvent:			HCN (1 stroke)
1,2-Propyl glycol	approx. 2.5 %	completely soluble	
Dipropyl glycol	10 %	completely soluble	> = 40
Triethylene glycol	8 %	completely soluble	approx. 30
Phenoxypropanols	10 %	completely soluble	approx. 30
Phenoxyethanol	10 %	completely soluble	approx. 30
1-Methoxy-2-propanol	10 %	completely soluble	approx. 30
Butyl diglycol	10 %	completely soluble	approx. 30
Ethylene glycol	<< 1 %	completely soluble	approx. 30
Deionized water	virtually insoluble (< 0.01 %)		

All solutions, after storage at room temperature for 2 months were without change, clear and slightly yellowish.

10 A 5 % strength aqueous dispersion of 3-thiocyanato-N-octylacrylamide is almost white, homogeneous and has an HCN emission of 13 ppm (5 strokes) (Dräger tube, see above).

Adding, for example, N-octylisothiazolone considerably decreased the HCN emission. The results were 15 roughly as for the organothiocyanate tested.

Example IX

Emission of thio compounds:

20 An aqueous Pyrion disulphide solution (containing 0.84 % active compound) was prepared from 7.46 g (0.02 mol) Pyrion-Na (40 % strength in water), 1 g (0.01 mol) of

sulphuric acid 96% and 1.18 g (0.011 mol) of H_2O_2 30% strength in 100 g of deionized water. It was clear and slightly yellow. A marked odour of thio compounds was noted.

5 If this solution was admixed with 10 g of a mixture of 5-chloro-N-methylisothiazolone and N-methylisothiazolone (approximately 14% by weight active compound content), the "thio odour" disappeared.

CLAIMS

1. Use of isothiazolone compounds, mercapto compounds, cyclodextrin compounds, iodopropynyl butylcarbamate,
5 diiodomethyl p-tolyl sulphone, zinc oxide, hydrogen peroxide or mixtures of two or more such compounds as additive for the reduction of emissions of volatile compounds which are potentially hazardous or cause unpleasant odour from compositions which comprise constituents which form such
10 compounds which are potentially hazardous or cause unpleasant odour.
2. Use according to Claim 1, characterized in that, as emission-reducing additive, use is made of
N-octylisothiazolone, 5-chloro-N-methylisothiazolone,
15 N-methylisothiazolone, a mixture of 5-chloro-N-methylisothiazolone and N-methylisothiazolone, 1,2-benzisothiazolone, 5,6-dihydro-2-methyl-2H-cyclopent(d)isothiazol-3(4H)-one, Pyrion disulphide and/or alpha-, beta- or gamma-cyclodextrin.
- 20 3. Use according to Claim 1 or 2, characterized in that, as emission-reducing additive, use is made of N-octylisothiazolone.
4. Use according to one of Claims 1 to 3, characterized in that the emission of HCN, amines or thio compounds and, in
25 particular, of HCN, is reduced.
5. Use according to Claim 4, characterized in that the emission of HCN is reduced from compositions which comprise compounds which have cyanide or thiocyanate ions or cyanide or thiocyanate substituents, in particular organothiocyanate
30 ions or organothiocyanate substituents.
6. Use according to one of the preceding claims, characterized in that the emission is reduced from liquid compositions, in particular from solutions and dispersions, preferably from aqueous dispersions.
- 35 7. Use according to Claim 6, characterized in that the emission is reduced from cleaning agents, disinfectants, preservatives, lubricants, impregnating agents, paints, coatings and plasters.

8. Use according to one of the preceding claims,
characterized in that the emission-reducing additive is used
at least in an amount of 0.01% by weight, preferably 0.01 to
10.0% by weight, in particular 0.1 to 7.5% by weight and
5 especially 0.5 to 5.0% by weight, based on the composition.